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Morphological, electrical and magnetic properties of BaAl$_2$Fe$_{12}$O$_{22}$ nano sized powders using chemical co-precipitation technique

Akant AK[1], Rathod UV[2], Giriya MN[3], Khobaragade CL[4*]

Author Affiliation:
[1]Research Scholar, Dept. of Appl. Physics, Manoharbhai Patel College of Engg & Technology, Gondia, India
[2]Research Scholar, Dept. of Appl. Physics, J. D. College of Engg Nagpur, India
[3]Asso. Professor, Dept. of Appl. Physics, Smt. Radhikatai Pandav College of Engg, Nagpur, India
[4]Asst. Professor, Dept. of Appl. Physics, Govindrao Wanjari College of Engg & Technology, Nagpur, India

*Corresponding author
Asst. Professor, Dept. of Appl. Physics, Govindrao Wanjari College of Engg & Technology, Nagpur,
India
Email: chanduparulk@gmail.com

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ABSTRACT

In the present investigation, the samples with chemical composition BaAl$_2$Fe$_{12}$O$_{22}$ have been synthesized using perfect stoichiometric proportions of nitrates by chemical co-precipitation technique. The pellets of 15 mm diameter were prepared and sintered at 1100℃ for 6 and 4 hrs and at 1000℃ for 6 hrs separately. The characteristic studies have been done using XRD, SEM, electrical and magnetic properties. XRD studies of the samples showed hexagonal Y-type structure with unit cell dimensions 'a' and 'c' varies from a = 5Å to 6Å and c = 43Å to 47Å pertaining to space group P6$_3$/mmc (No. 194). The variation in the values of lattice parameter has to be recorded with increase in Mn-Zn conc. The magnetic properties of prepared Y-type Ca-hexaferrite powder were investigated by VSM studies at 15,000 Gauss magnetic field. The transition temperature and activation energies have been investigated from electrical behavior of the samples.

Keywords: XRD, SEM, VSM, Electrical and Magnetic Properties, Curie temperature.

1. INTRODUCTION

Hexaferrites have been intensive studies due to a combination of good magnetic properties and low cost. This large family of oxides with hexagonal crystal structure contains ferrimagnetic compounds with easy axis of magnetization (e.g. M-type ferrites) and easy plane of magnetization (e.g. Y-type ferrites). Hence, hex ferrites have been widely adopted in two distinct fields: Permanent magnets and microwave technology components (Raj et al., 1995). On the other hand, Y-type ferrites BaAl$_2$Fe$_{12}$O$_{22}$ can undergo spin reorientation transitions (SRT) between different anisotropy configurations (easy plane ↔ easy cone ↔ easy axis) induced by change of temperature or applied magnetic field (Vishwanathan and Moorthy, 1990; Verwey and De Boer, 1936; Koops, 1951; Subramanian and Marks, 2004; Smith et al., 1968; Mukhtar et al, 2013; Cullity, 1976; Kamba et al., 2010). The transition temperature can be tuned by modifying the chemical composition (substitution of divalent and trivalent metal ions). Moreover, some potential

application of Y-type hexaferrites at 1100℃ & 1000℃ temperatures have been such as magnetic recording media, permanent magnets, magnetic yokes, latches, phase shifter etc. (Smith et al., 1968; Mukhtar et al, 2013).

Conventional ceramic method of hexaferrite synthesis is efficient, but requires elevated temperatures for solid state reaction to occur between premixed powders. Alternative production routes (aerosol pyrolysis, chemical co-precipitation, glass crystallization, hydrothermal synthesis, etc.) are intended to improve mixing of initial components down to atomic level and thereby to facilitate diffusion. In particular, a chemical co-precipitation technique enables to obtain sufficiently homogeneous precursors for low temperature synthesis of nanosized ferrites. However, hexagonal ferrites with complex layered crystal structures still require relatively high temperatures to form because of thermodynamic stability conditions. Trying to avoid rapid growth of grains, we have used this soft-chemistry approach for production of Y-type ferrites. An important goal was to determine the heat treatment regimens ensuring complete transformation of a precursor into the smallest particles of single Y-phase. In this paper the results of morphological, electrical, magnetic and mechanical characterization $BaAl_2Fe_{12}O_{22}$ of powders are presented.

2. MATERIALS AND METHODS

Hexaferrite powders have been prepared by a chemical co-precipitation method (Satone et al., 2014). The A. R. grade nitrates have been used as starting material. Stoichiometric proportion of barium nitrate, ferric nitrate, aluminium nitrate (99.9%) etc. have been dissolved one by one in 100 ml of de-ionized water. Ammonia solutions (30%) have been added slowly in the mixture to adjust pH of 8. The mixed solutions have been stirred for 2 hr and will be kept at room temperature for 24 hr for aging. The calcium hexaferrites precipitates have been separated in a centrifuge machine at 2500 rpm for 20 minute. The prepared precipitates have been washed in 1:1 mixture of methanol and acetone followed by 100% de-ionized water to remove impurities. The precipitates have been dried at 100℃ for 24 hrs and calcinated at 1000℃ & 1100℃ for 4 hrs and 6 hrs separately to obtain $BaAl_2Fe_{12}O_{22}$ hexaferrite particles (Jotania et al., 2009).

Crystal structure has been examined by PA Analytical X'Pert Pro X-ray diffraction (XRD) in Cu-Kα radiation with accelerator detector for rapid data acquisition. To ensure the single-phase formation of the hexagonal structure, X-ray studies have been carried out. Magnetization curves were recorded on Lake Shore vibrating sample magnetometer (VSM). The lattice parameters and X-ray densities of the samples have been computed from XRD analysis. The bulk density has been determined by mass and bulk volume of the samples, while porosity for each sample has been calculated separately. Structural analysis has been carried out from SEM and XRD of the studied materials.

3. RESULTS

Morphological Analysis

The BaY-hexaferrites ($BaAl_2Fe_{12}O_{22}$) with substitution of aluminium (Al) calcinated at temperatures at 1100℃ for 6 and 4 hrs and at 1000℃ for 6 hrs separately. The crystallographic results described (Table-1) and the XRD pattern (Figure 1) reveals the hexagonal crystal structure and most of the lines can unequivocally be indexed for the magnetoplumbite structure. All the three members of series with the values 'a' and 'c' and cell volume varies in the range of a = 5.85Å to 5.84Å and c = 44.13Å to 46.11Å and cell volume V = 1309.90 Å^3 to 1365.31 Å^3 (Table-1). However, these values are analogous to the values of a = 5.87Å, c = 43.29Å for $Ba_2Mn_2Fe_{12}O_{22}$ calcinated at 900℃ to 1100℃ as mentioned in (Mukhtar et al, 2013). This variation in the value of 'a' and 'c' is obvious as ionic radii of Al^{3+}(0.535Å) is smaller as compared to Ba^{2+}(1.35Å) than Fe^{3+}(0.645Å) ions which has replaced Fe^{3+} ions i.e. atoms shrinks towards a and c axis.

From the SEM photograph (Figure 2(a), 2(b), 2(c)), the average grain size of BaAl$_2$-Y ferrites is geometrically estimated. The particle size varies in the range of 129.77 nm to 59.13 nm for the calcination's temperature from 1100℃ to 1000℃ for time interval of 6 hours and 129.33nm to 65nm for the calcinations temperature of 1100℃ for time interval of 6 to 4 hours. It means that the sizes of the molecules of the samples are observed in nano range which are hexagonal in shape and also it is concluded from the calculated values of XRD analysis (Kumar et al., 2015).

The bulk density of the compounds of these series is observed to be decreased with the decreasing calcinations temperature from 1100℃ to 1000℃ for time interval of 6 hours and also for the calcinations temperature of 1100℃ for time interval of 6 to 4 hours, which may be due to contraction of grain size due to decrease of the calcinations temperature with time. The X-ray density (dx) are calculated, which depends on the molecular weight and the volume of the samples. The bulk density (d_B) of the sample has been calculated from the geometry and the mass of the samples. The percentage porosity (P) of the samples has been calculated from X-ray density and bulk density, which shows rising trend with decreasing calcinations temperature (Table-1).

Table 1 Ba-Y hexaferrite calcinated at different temp for 4 and 6 hours showing lattice constants, particle size, bulk density, X-ray density, porosity, molecular wt and Curie temp.

| Sample | Calcination Temp (°C) | Time (Hrs) | Lattice Constant | | | Volume | Particle size | Bulk density | X-ray Density | Porosity | Molecular wt | Curie Temp. |
			a (Å)	c (Å)	c/a	V(Å³)	D (nm)	d_B (gm/cm³)	d_x (gm/cm³)		(gm/mole)	(T_c)(K)
$BaAl_2Fe_{12}O_{22}$	1100	6	5.85	44.13	7.5	1309.87	129.77	2.469	4.6143	0.465	1213.49	478
$BaAl_2Fe_{12}O_{22}$	1100	4	5.86	44.44	7.5	1325.05	65	2.095	4.4614	0.5304	1213.49	448
$BaAl_2Fe_{12}O_{22}$	1000	6	5.84	46.11	7.8	1365.27	59.13	2.045	4.4270	0.538	1213.49	448
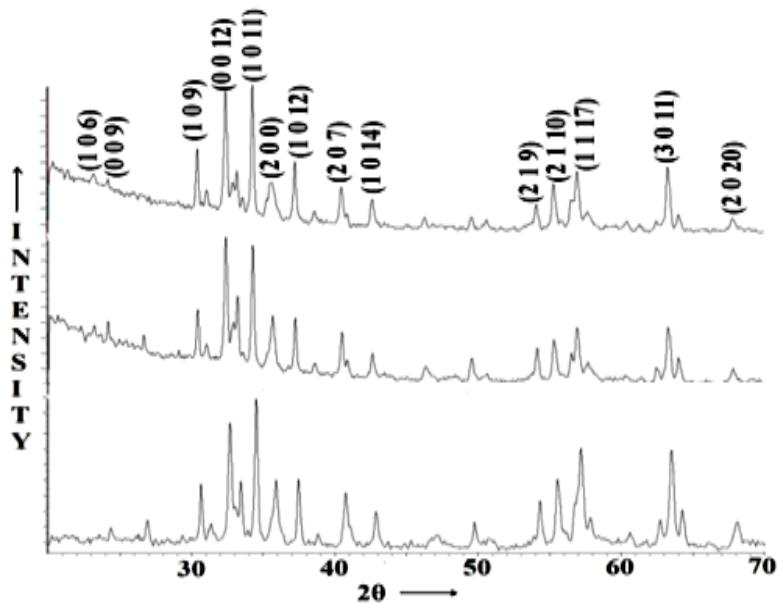
**Figure 1** The XRD of Ba-Y hexaferrite compounds calcinated at different temp for 4 and 6 hours
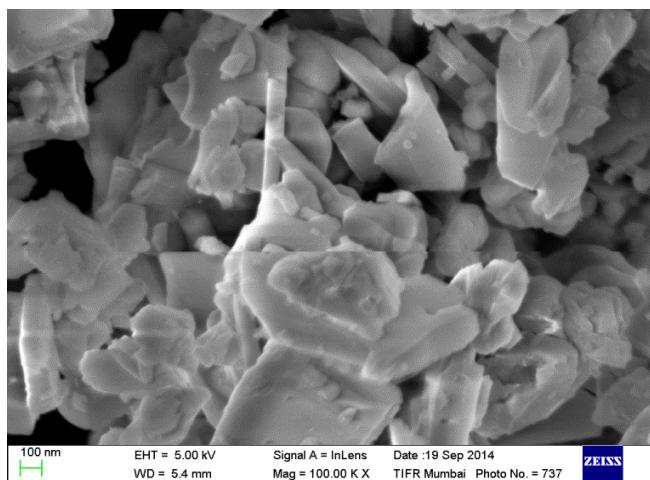
**Figure 2(a)** $BaAl_2Fe_{12}O_{22}$ calcinated at 1100⁰C for 6 Hrs (*Source*: Rathod et al., 2014).
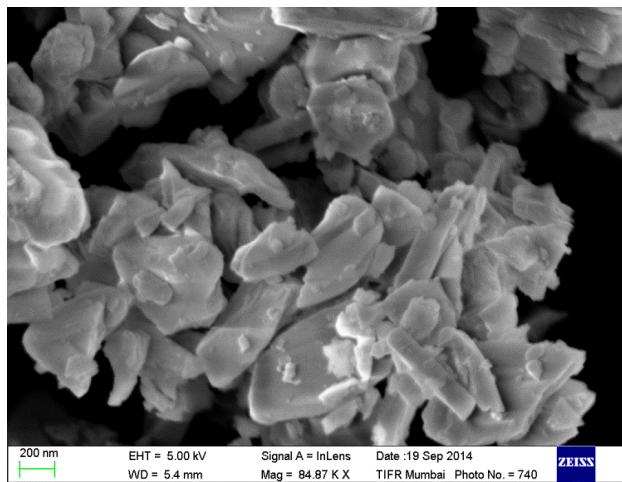
Figure 2(b) $BaAl_2Fe_{12}O_{22}$ calcinated at 1100^0C for 4 Hrs (*Source*: Rathod et al. 2014).
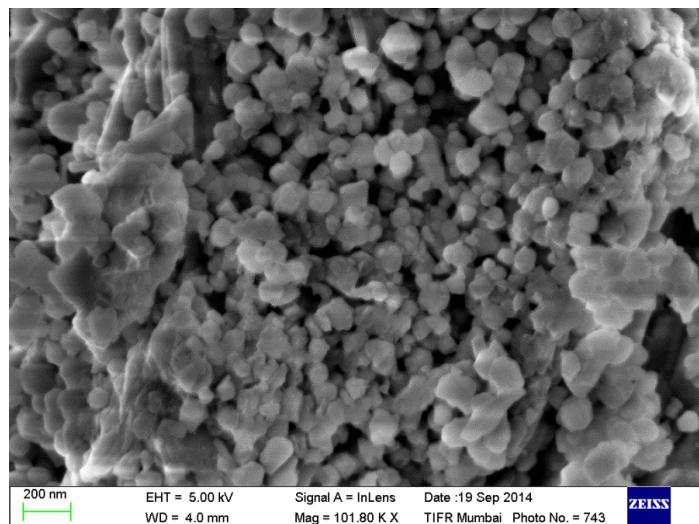
**Figure 2(c)** $BaAl_2Fe_{12}O_{22}$ calcinated at 1000^0C for 6 Hrs (*Source*: Rathod et al., 2014).

The X-ray density has been calculated using the relation for hexagonal ferrite Moinuddin and Murthy, (1993) given below:

$$dx = \frac{2M}{N\ 0.866a^2c}$$

where, M = molecular weight of the sample, volume (V) for hexagonal = $0.866a^2c$, N = Avogadro's number & Lattice parameters is a, c.

The X-ray density (dx) depends on the lattice constant and molecular weight of the samples whereas the bulk density (d_B) of the sample can be calculated from the geometry of the crystals and mass of the samples.

The well-known Scherer formula has been used to determine the particle size from the line broadening of diffraction profile of the strongest peak. The formula, excluding the effects of the machine broadening to minimize errors, is given below:

$$D = \frac{k\lambda}{b\ \cos\theta}$$

where, D = average particle size, k = Scherrer constant (k = 0.9), λ = wavelength of radiation (1.54056 Å), b = peak with at half height [full width half maxima (FWHM)] and θ corresponds to the peak position measured in radians. It shows that the particle size varies in the range from 129.77 nm to 59.13 nm. It means that the size of the particle is very small and they are closely bound with each other in the crystal structure. Therefore, less porosity is found in these compounds.

In order to monitor the structural changes during the synthesis process, FTIR spectra of dried compounds of this series have been taken within wave number range from 4000cm^{-1} to 400cm^{-1} (Figure 3(a), 3(b), 3(c)). The spectra of dried compounds for BaAl$_2$Y-Hexaferrites have been shown in (Table-2), indicates the characteristics bands in the range 453.27 cm^{-1} to 599.86 cm^{-1}which attributes to Ba-O-Al stretching vibration of the BaAl$_2$-Y Hexaferrites (Hongguo et al., 2000).

Table 2 Ba-Y hexaferrite sample shows absorption peaks of FTIR spectra.

Sample	Calcination Temp (℃)	Time (Hrs)	Absorption Peaks	
			Upper peak cm^{-1}	Lower peak cm^{-1}
BaAl$_2$Fe$_{12}$O$_{22}$	1100	6	597.93	462.92
BaAl$_2$Fe$_{12}$O$_{22}$	1100	4	599.86	482.20
BaAl$_2$Fe$_{12}$O$_{22}$	1000	6	590.22	453.27
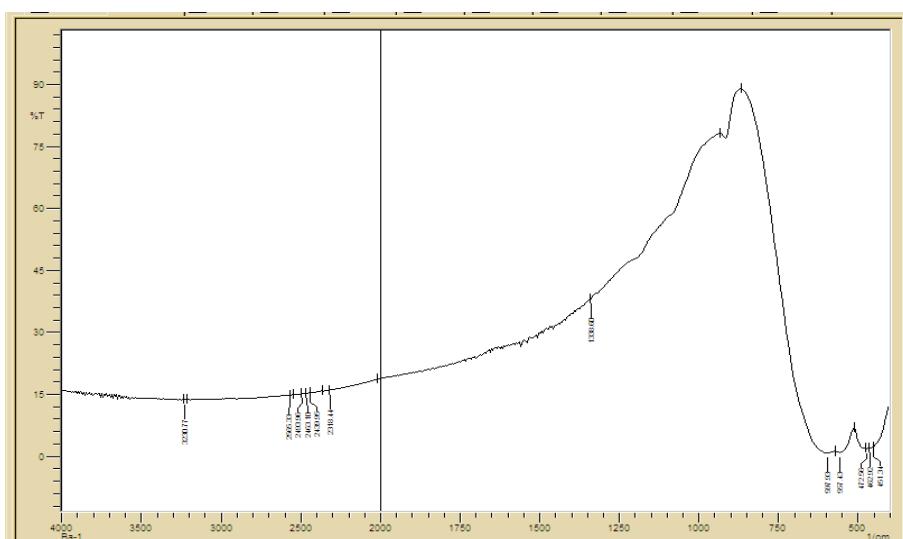
Figure 3(a) FTIR spectra of BaAl$_2$Fe$_{12}$O$_{22}$ calcinated at 1100^{0}C for 6 Hrs
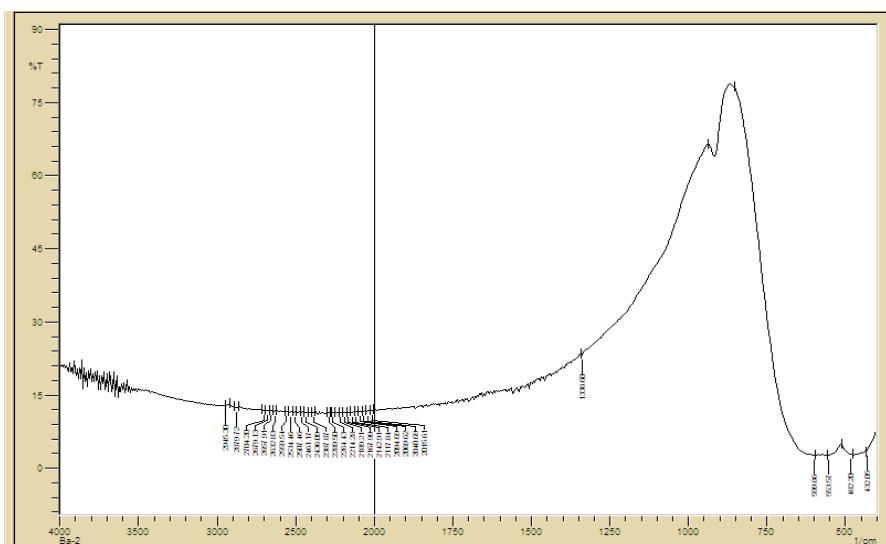
Figure 3(b) FTIR spectra of BaAl$_2$Fe$_{12}$O$_{22}$ calcinated at 1100^{0}C for 4 Hrs
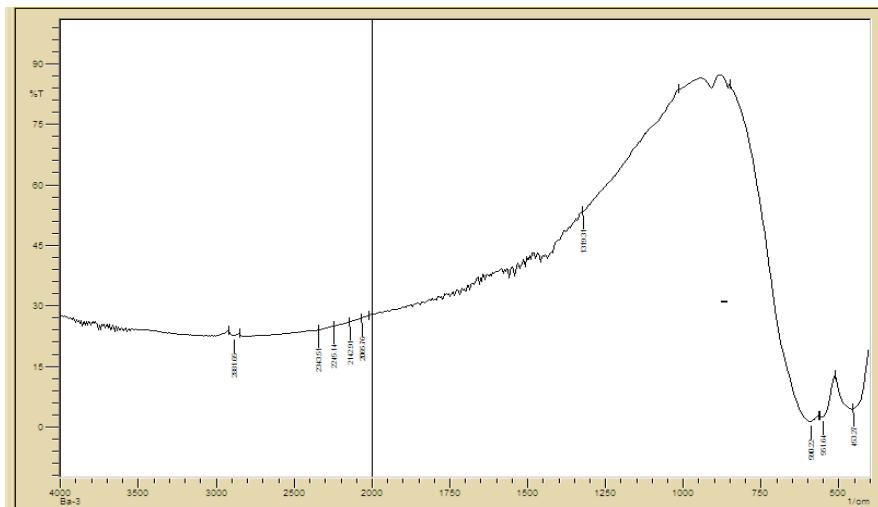
Figure 3(c) FTIR spectra of BaAl$_2$Fe$_{12}$O$_{22}$ calcinated at 1000^{0}C for 6 Hrs

Electrical Properties

The electrical studies of the samples under study were measured by applying dc voltage. The electrical conductivity and dielectric measurement are done by two probe method in the temperature range 303 K to 703 K. The plot of logarithmic electric conductivity versus inverse of temperature shows changes in slope in the neighborhood of Curie temperature (T$_c$). The activation energy below and above the Curie temperature was calculated separately. It has been observed that the activation energy is less in ferrimagnetic and more in paramagnetic region, which indicates semiconducting nature of the compounds (Mukhtar et al., 2013).

The electrical resistivity-temperature behaviour of the sample obeys the Willson's law

$$\rho = \rho_o exp(\frac{\Delta E}{KT})$$

It has been seen from the (Figure 4(a), 4(b), 4(c)) that the transition temperature (T$_t$) at which the kinks in the plot of log (σ) versus 1/T was observed in the specimens under investigations is in the neighborhood of its Curie temperature (T$_c$). This reveal that the kinks observed in Aluminium substituted Ba-Y ferrites have been attributed to the magnetic transition, observed a minute change in slope of Curie temperature in the plot of log (ρ) Vs 10^3/T for Ba$_2$Mn$_2$Fe$_{12}$O$_{22}$ as we observed in the present investigation of Ba-Y hexaferrites (Mukhtar Ahmad et al., 2013). The electrical conductivity results obtained at room temperature are given in (Table-3) and logarithmic conductivity (logσ) versus inverse of temperature (1/T) plots has been shown in (Figure 4(a), 4(b), 4(c)). It is evident that the value of electrical conductivity at room temperature varies in the ranges of 0.499 x 10^{-6} to 15.22 x 10^{-6}Ω^{-1}.cm^{-1}.

The dielectric measurements have been carried out over the frequency range 100Hz to 9.9MHz at room temperature using Weyn Kerr precision impendence Analyser 6500B. The variation of dielectric loss tangent tan (δ) with the log (F) for the series of compounds (Figure 5(a), 5(b), 5(c)). It clears from the figure that the dielectric loss tangent (tanδ) decreases with the increasing measuring frequency.

Table 3 Ba-Y hexaferrite compounds shows conductivity and activation energies in ferri a para magnetic region.

Sample	Calcination Temp (°C)	Time (Hrs)	Room temp. Resistivity(ρ) (Ω.cm)	Room temp. Conductivity(σ) (Ω.cm)$^{-1}$	Activation Energy ΔE	
					Ferri (eV)	Para (eV)
BaAl$_2$Fe$_{12}$O$_{22}$	1100	6	20.03 x10^5	0.499x10^{-6}	0.374	0.404
BaAl$_2$Fe$_{12}$O$_{22}$	1100	4	4.6 x10^5	2.17x10^{-6}	0.497	0.524
BaAl$_2$Fe$_{12}$O$_{22}$	1000	6	0.657 x10^5	15.22x10^{-6}	0.593	0.667
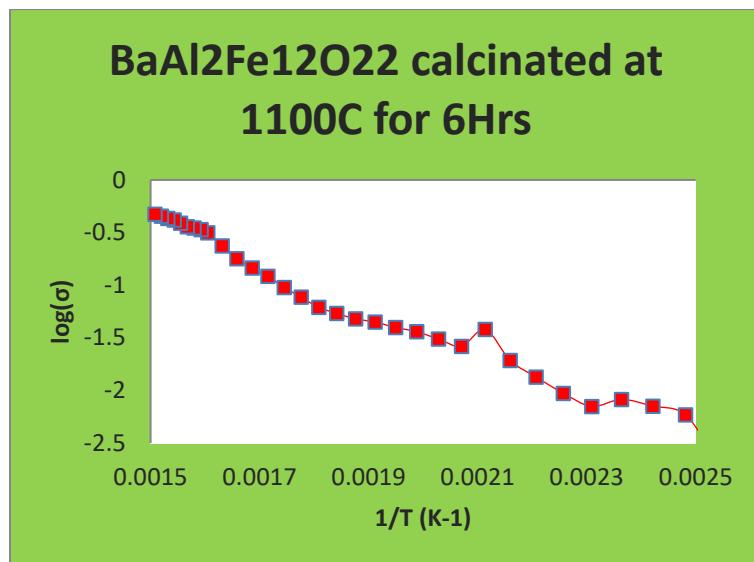
Figure 4(a) Plot of log (σ) versus 1/T of BaAl₂Fe₁₂O₂₂ calcinated at 1100°C for 6 Hrs
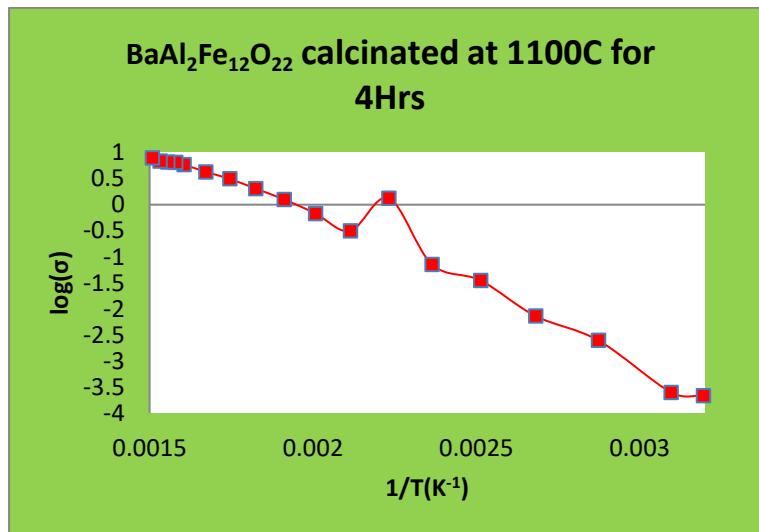

**Figure 4(b)** Plot of log (σ) versus 1/T of BaAl₂Fe₁₂O₂₂ calcinated at 1100°C for 4 Hrs.
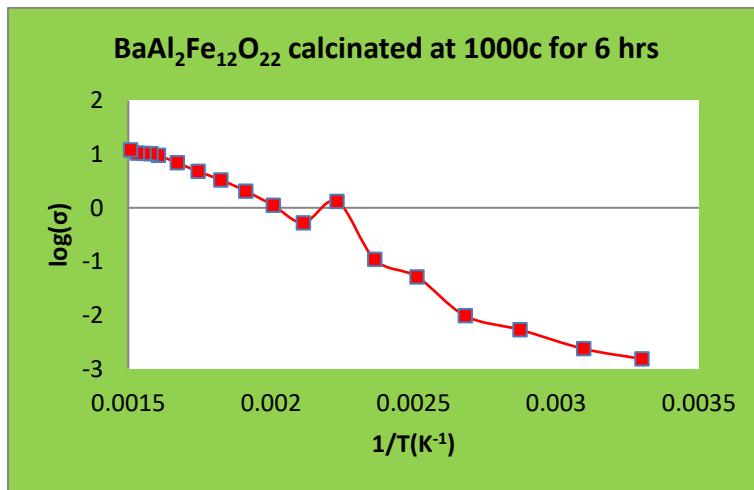
**Figure 4(c)** Plot of log (σ) versus 1/T of BaAl₂Fe₁₂O₂₂ calcinated at 1000°C for 6 Hrs
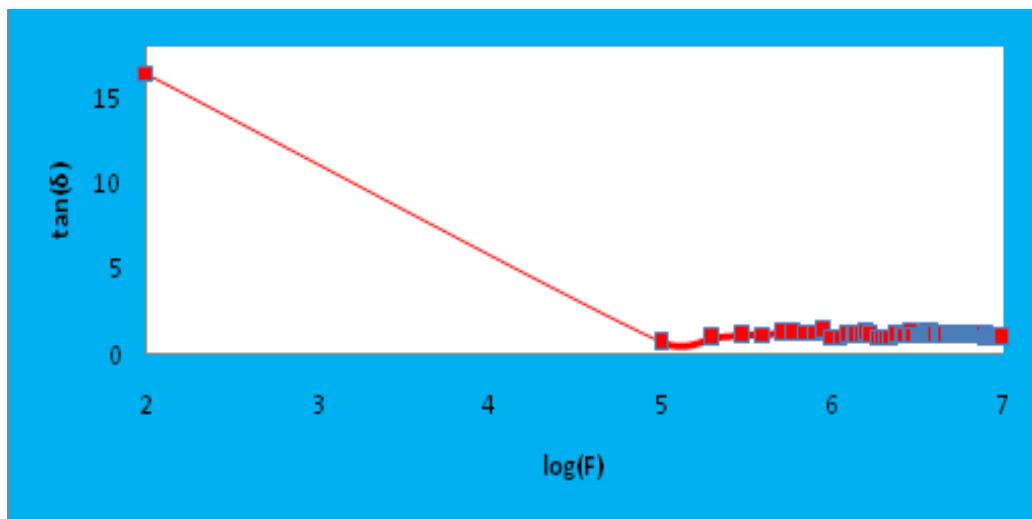
Figure 5(a) Plot of tan (δ) versus log (F) of $BaAl_2Fe_{12}O_{22}$ calcinated at 1100°C for 6 Hrs
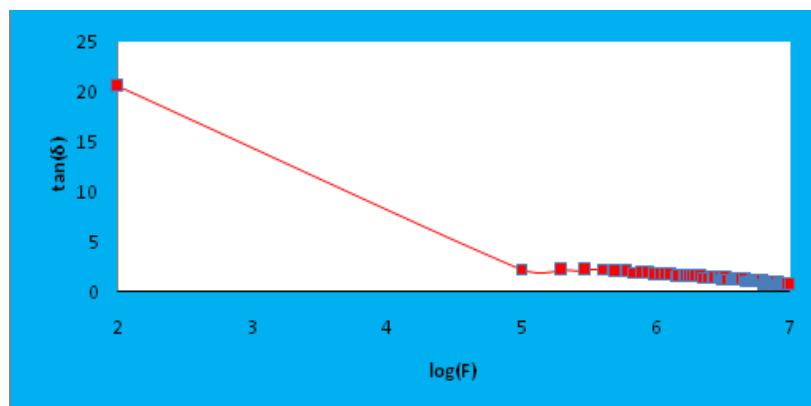
Figure 5(b) Plot of tan (δ) versus log (F) of $BaAl_2Fe_{12}O_{22}$ calcinated at 1100°C for 4 Hrs
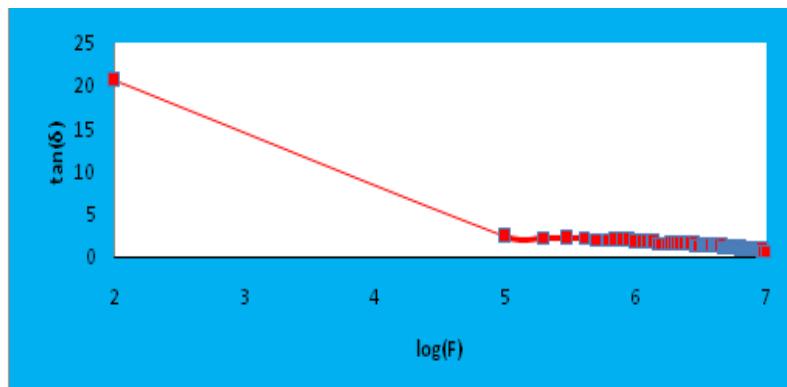
**Figure 5(c)** Plot of tan (δ) versus log (F) of $BaAl_2Fe_{12}O_{22}$ calcinated at 1000°C for 6 Hrs

Magnetic Properties

The B-H curve measurement have been carried out in the compounds which are calcinated at 1100°C and 1000°C for 6 hours in order to study its coercivity, remanance, saturation magnetization and squareness ratio from these values magnetic moments were calculated.

The hysteresis loops have been traced at varying external applied magnetic field of 15000 G, volume of the sample has been taken about 31 to 35 mg. The shape and size of hysteresis loop obtained for these two compounds (Figure 6(a), 6(b)). If we compare the size and shape of compounds, it has been observed that Ba-Y hexaferrites calcinated at 1100°C for 6 hours is more magnetic in nature than Ba-Y hexaferrites calcinated at 1000°C for 6 hours. The above observation is corroborated with the evidence showing larger value of coercivity, remanance and saturation magnetization for Ba-Y hexaferrites calcinated at 1100°C for 6 hours. It has

been observed from the result that for Ba-Y hexaferrite, saturation magnetization (M_s) increase but remanance (M_r) decreases, as calcinations temperature decreases from 1100°C to 1000°C shown in (Table-4). Same trend is observed in other hexaferrites, calcinated at different higher temperatures for $Ba_2Zn_2Fe_{12}O_{22}$ (Fang et al., 2008). As hysteresis study has been carried out at the external magnetic field of 15000 G, the compounds showing a nature of soft ferrites however at higher magnetic field, it has been showing higher values of coercivity, remanance and saturation magnetization.

Table 4 The Ba-Y hexaferrite sample shows coercivity, remanance, saturation magnetization and squareness ratio from VSM of each sample.

Sample	Calcination Temp (°C)	Time (Hrs)	Coercivity (H_c) (Gauss)	Remanence (M_r) (emu/gm)	Saturation Magnetization (M_s) (emu/ gm)	Square-ness Ratio M_r/M_s
$BaAl_2Fe_{12}O_{22}$	1100	6	1877.8	0.1444	0.22431	0.6439
$BaAl_2Fe_{12}O_{22}$	1000	6	1149	0.119	0.22742	0.5233
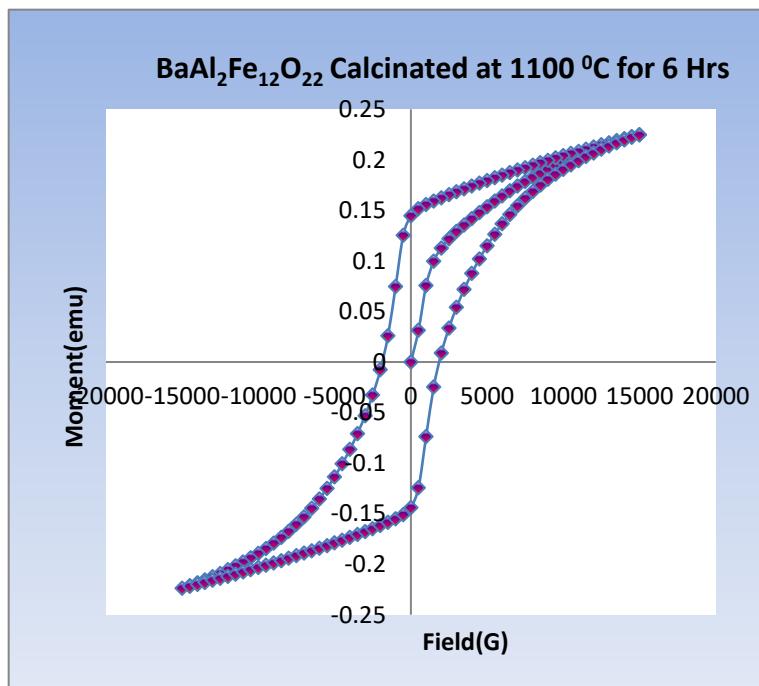
Figure 6(a) VSM of $BaAl_2Fe_{12}O_{22}$ compound calcinated at 1100°C for 6 Hrs
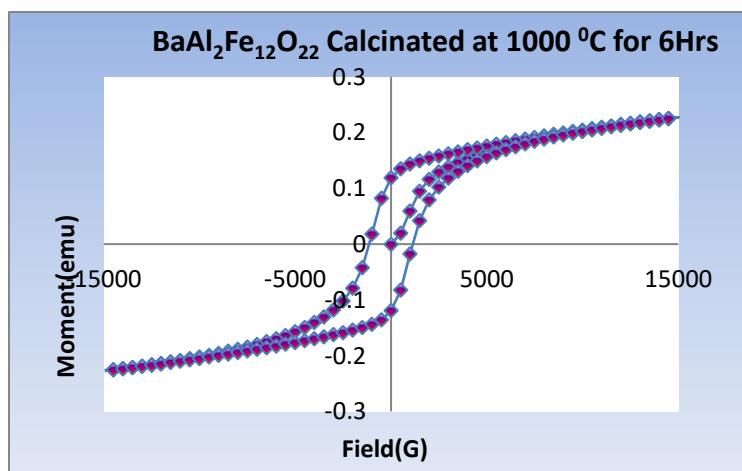

Figure 6(b) VSM of $BaAl_2Fe_{12}O_{22}$ compound calcinated at 1000°C for 6 Hrs

4. DISCUSSION

The variation in the value of 'a' and 'c' is obvious as ionic radii of Al^{3+}(0.535Å) is smaller as compared to Ba^{2+}(1.35Å) than Fe^{3+}(0.645Å) ions which has replaced Fe^{3+} ions i.e. atoms shrinks towards a and c axis. It is also found that, almost all the reflection of the reported compounds has been indexed in our compounds. The exact matching of crystallographic data of the above sample to that of reported one confirms the formation of all the samples in a single homogeneous phase with hexagonal structure (Cullity, 1976). It has been observed that there were no significant changes in the crystal lattice of Aluminium substituted Ba-Y hexaferrites found with changing calcinations temperature from 1100℃ to 100℃ within time interval of 6 hours to 4 hours. These barium ions with ionic radius (1.35Å) are slight smaller than the oxygen ions with ionic radius (1.4Å). Therefore the distance between the oxygen layers which contain no barium ion are 2.40Å and 2.32Å respectively. The projection of the distance between the centers of the Ba ions on to the axis is 2.90Å, from which it follows that the centers of the barium ions lies at a distance of 0.25Å out of the plane of the nearest oxygen layer (Jotania and Patel, 2012).

The SEM results reveal that the grain size varies in the range of 129.77 nm to 59.13 nm for the calcinations temperature from 1100℃ to 1000℃ for time interval of 6 hours and 129.33nm to 65nm for the calcinations temperature of 1100℃ for time interval of 6 to 4 hours. It means that the sizes of the molecules of the samples are observed in nano range which are hexagonal in shape and also it is concluded from the calculated values of XRD analysis (Kumar et al., 2015). The bulk density of the compounds of these series is observed to be decreased with the decreasing calcinations temperature from 1100℃ to 1000℃ for time interval of 6 hours and also for the calcinations temperature of 1100℃ for time interval of 6 to 4 hours, which may be due to contraction of grain size due to decrease of the calcinations temperature with time. Other reason might be the modification may be due to difference in the specific gravity of compositional oxides that may be affected with changing calcinations temperature.

The X-ray density (dx) depends on the molecular weight and the volume of the samples. The bulk density (d_B) of the sample has been calculated from the geometry and the mass of the samples. The percentage porosity (P) of the samples has been calculated from X-ray density and bulk density, which shows rising trend with decreasing calcinations temperature. This is due to the fact that value of lattice parameter gets altered by decreasing the calcinations temperature which leads to increase the porosity for Ba-Y hexaferrites. In this context another reason might be difference in the melting point of the reacting oxides used. The numbers of the pores get reduced at higher temperature at 1100℃ as a result of which the individual grains come closer to each other and effective cross sectional area of grains to grains contact increases. This in turn results in greater densification and less porosity at higher temperature (Moinuddin and Murthy, 1993).

The particle size of the studied compound varies in the range from 129.77 nm to 59.13 nm (Table 1). It means that the size of the particle is very small and they are closely bound with each other in the crystal structure. Therefore less porosity is found in these compounds. The FTIR spectra of dried compounds of this series are taken within wave number range from 4000cm^{-1} to 400cm^{-1}. The spectra of dried compounds for BaAl₂Y-Hexaferrites indicates the characteristics bands in the range 453.27 cm^{-1} to 599.86 cm^{-1} which attributes to Ba-O-Al stretching vibration of the BaAl₂-Y-hexaferrites (Hongguo et al., 2000).

The plot of logarithmic electric conductivity versus inverse of temperature shows changes in slope in the neighborhood of Curie temperature (T_c). The activation energy below and above the Curie temperature has been calculated separately. It has been observed that the activation energy is less in ferrimagnetic and more in paramagnetic region. The plots are linear indicating any absence of impurity in crystal lattice. An increase of the temperature from room temperature, the resistivity of the sample decreases for each of compound in the Ba-Y series of the hexaferrites, which attributes directly about the tight bonding of ions at room temperature at their interstitial sites, which originates the hopping mechanism in the crystal lattice. The activation energy was found to be decreasing with the decreasing calcinations temperature. The conduction mechanism may be explained on the basis of Koop's hopping mechanism of the localized d-electron model for Y-Type hexaferrites (Koops, 1951).

This decrease behaviour of dielectric loss tangent (tan δ) with the increasing frequency has been explained on the basis of the assumption that is made in the mechanism of polarization process in ferrites. The behaviour of tan δ with the frequency shows relaxation peaks which shift towards lower frequencies with decreasing calcinations temperature from 1100℃ to 1000℃ (Nandotaria and Jotania, 2011). It has been observed from the result that for Ba-Y hexaferrite saturation magnetization (M_s) increase but remanance (M_r) decreases, as calcinations temperature decreases from 1100℃ to 1000℃. These modifications in the values has been observed due to Fe-O-Fe exchange interaction and reduced in lattice dimension site which directly affects on site distribution, Curie constant and saturation magnetization. The temperature dependence of magnetization can be explain in light of cationic distribution and lattice dislocation among the available tetrahedral and octahedral sites in spinal and garnet site(Sharma et al., 2008).

5. CONCLUSION

Y-type barium hexaferrite have been synthesized by chemical co-precipitation method using nitrates. The obtained products exhibit well crystalline phase of $BaAl_2Fe_{12}O_{22}$ typical hexagonal structure. The magnetic measurement for the calcined Y-type hexaferrite was observed and the values are compared with previous research reports, which exhibits enhanced hard magnetic property due to the reduced particle size. The electrical conductivity measurements observed Curie temperature in the range 448K to 478K. The activation energies have been found in the range of 0.374 eV to 0.593 eV in ferrimagnetic and 0.404 eV to 0.667 eV in paramagnetic region.

Summary of research

The lattice parameter 'a' shows the decreasing trend and 'c' values shows increasing trend with the added impurities this is due to the difference between ionic radii of Al^{3+}(0.535Å) is smaller as compared to Ba^{2+}(1.35Å) than Fe^{3+}(0.645Å) ions which has replaced Fe^{3+} ions i.e. atoms shrinks towards a and c axis. The particle size for the studied samples have been calculated and found in the range of 59.13 nm to 129.77 nm shown in (Table 1). It has been reported that the crystallite size less than 50 nm is required in obtaining the suitable signal-to-noise ratio in the high density recording media.

The Curie temperature (Tc) has been found in the range of 448K to 478 K. The activation energy has been found in the range of 0.374 eV to 0.593 eV in ferrimagnetic and 0.404 eV to 0.667 eV in paramagnetic region. The saturation magnetization carried out 0.22431emu/g at 1100℃ & 0.22742emu/g at 1000℃, coercivity is 1877.8 G at 1100℃ & 1149 G at 1000℃, remanance of magnetization is 0.1444emu/g at 1100℃ & 0.119emu/g, squareness ratio (Mr/Ms) is 0.6439 at 1100℃ & 0.5233 at 1000℃. It is well known that the coercivity of barium hexaferrite depends on many factors, such as chemical composition, particle size, degree of crystallinity, microstructure, magnetic anisotropy, etc.

Future issues

I believe that many scientists doing work on Y-type ferrite. To pay attention to the real research on ferrite data obtained from last 20 years and summarized a little in this work. The main application of this developed material is in high density recording media in obtaining suitable signal-to-noise ratio.

Ethical approval

Not applicable.

Informed consent

Not applicable.

Conflicts of interests

The authors declare that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

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